The effect of high pressure processing on the crystallinity of polyethylene films tested by differential scanning calorimetry and X-ray diffraction methods

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ABSTRACT

This study investigated the effect of high pressure processing (HPP) on the crystallinity of polymeric films used for food packaging. This information was needed because a gap in knowledge exist in this area.

The objective of this study was to investigate the effects of HPP on the morphology of low-density polyethylene (LDPE) by monitoring the crystallinity of the films before and after the treatments.

Pouches made from the PE films were filled with 95% ethanol as a food simulant, then high pressure processed at 200, 400, 600 and 800 MPa for 5 and 10 minutes at 25 and 75˚C. The pouches were then opened, the liquid empted and the pouches dried. Control samples were similarly prepared but not high pressure processed. The crystallinity of the film samples were then measured using differential scanning calorimetry (DSC) and X-ray diffraction testing methods.

Results of the DSC experiments showed that crystallinity changes were not detectible. However, the X-ray diffraction method showed significant (*P<0.05*) crystallinity increases. These were: 1) 2.37 % for HPP temperature change from 25 to 75˚C; 2) 1.15 % for HPP time change from 5 to 10 minutes; 3) 14.91% for HPP intensity change from 0.1 MPa (control) up to 800 MPa.

These findings allowed us to better anticipate the behavior of LDPE films used to package high pressure processed foods.

INTRODUCTION

High pressure processing denatures protein molecules whereas many compounds responsible for sensory and nutritional quality are unaffected. Thus, certain foods could be safely high pressure processed to produce products with more "natural" taste and with higher nutrient content when compared to the same foods treated by thermal processing.

The availability of flexible plastic films has allowed the application of HPP to pre-packaged foods. Most of these films are capable of withstanding the high pressure process without visible signs of integrity loss. However, HPP has the potential to produce undesirable effects in some polymeric films. Limited studies have shown that some flexible materials can loose significant barrier properties to oxygen, carbon dioxide and water vapor when exposed to HPP. However, there is limited information of the effects of HPP on the crystallinity of polyethylene. Changes to the crystallinity of a polymer can affect its gas and vapor permeability, stiffness and thermal properties, for example. It is thus imperative to gain a clear understanding of which materials are affected by HPP and how these changes can be detected.

This study will provide important scientific information that is essential for a better understanding of the morphological changes to polymers caused by HPP. Therefore, this information could be used to help synthesize and/or modify polymers that are best suited for HPP.

MATERIALS & METHODS

Sample preparation

Duplicate sample pouches were made from LDPE films measuring 10 cm × 10 cm. They were filled with 95% ethanol as a food simulant, sealed without headspace using a KF Model AIE-300 impulse sealer (120V, 60 Hz), then high pressure processed.

High pressure processing (HPP)

The HPP was performed with a QFP-6 Quintus high pressure food processor. The HPP conditions were 200, 400, 600 and 800 MPa for 5 and 10 minutes at 25 and 75°C. Control samples were similarly prepared but not high pressure processed.

Differential scanning calorimetry (DSC)

A Model 2920 TA Instruments modulated differential scanning calorimeter (DSC) (New castle, DE), equipped with a data collection station, was used to investigate how HPP affected the thermal transition of the LDPE. This was done at a heating rate of 5°C/min from 25 to 150°C. The weight of each sample was 9 mg. Thermal parameters such as melt temperature (Tm) and heat of fusion (△H) were determined from transitions in the DSC thermograms. The crystallinity was calculated from the ratio of the samples' △H to 288 J/g (△H for 100% crystalline LDPE).

X-ray diffraction (XRD)

X-ray diffraction patterns for the LDPE films were obtained using an X-ray diffractometer DMS 2000 Scintag Series. The X-ray copper target tube was operated at 40kV and 30mA. The X-rays passed through a 1° divergence slit onto samples of the films placed in the specimen chamber. The diffracted radiation from the samples passed through a 0.1° scatter slit then reached the monochromator. All the diffraction patterns were examined at room temperature and under constant operation condition.

RESULTS

Figure 1. The effect of HPP on *the crystallinity* at different conditions (temperature and time) by DSC

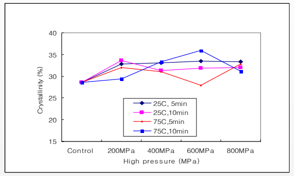


Figure 2. The effect of HPP on *the melt temperature* at different conditions (temperature and time) by DSC

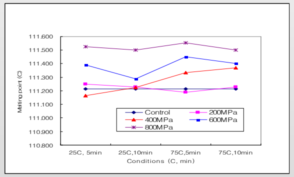
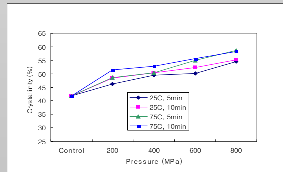


Figure 3. The effect of HPP on *the crystallinity* at different conditions (temperature and time) by X-ray diffraction



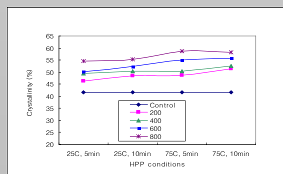
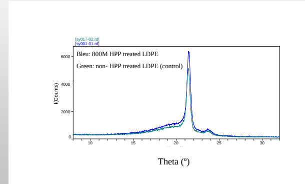


Figure 4. Thermograms of non-HPP treated film (control) and HPP treated film (800MPa, 75°C, 10min) by X-ray diffraction



CONCLUSIONS

1. HPP caused significant changes to the crystallinity of LDPE.

2. HPP caused an increase in the melt temperature of the HPP treated samples.

3. The X-ray diffraction method successfully detected the crystallinity changes. These were: 1) 2.37 % for HPP temperature change from 25 to 75˚C; 2) 1.15 % for HPP time change from 5 to 10 minutes; 3) 14.91% for HPP intensity change from 0.1 MPa (control) up to 800 MPa.

4. The DSC analyses showed no detectible change in the enthalpy of fusion and crystallinity of the material after HPP up to 800 MPa (P*<0.05*). However, DSC showed changes in the melt temperature.

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